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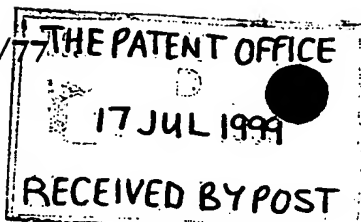
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The Patent Office

Cardiff Road
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Gwent NP9 1RH

1. Your reference

MPD318/GB/RGMS

2. Patent application number

(The Patent Office will fill in this part)

9916744.7

17 JUL 1999

3. Full name, address and postcode of the or of each applicant (underline all surnames)

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Patents ADP number (if you know it)

6804264002

If the applicant is a corporate body, give the country/state of its incorporation

ENGLAND

4. Title of the invention

STRUCTURED SURFACTANT SYSTEMS

5. Name of your agent (if you have one)

R G M SAVIDGE

"Address for service" in the United Kingdom to which all correspondence should be sent

(including the postcode)

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Patents ADP number (if you know it)

FS1/77 818/2000 HC

5249004

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number
(if you know it)

Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

NO

- a) any applicant named in part 3 is not an inventor, or
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Patents Form 1/77

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Priority documents	NONE
Translations of priority documents	NONE
Statement of inventorship and right to grant of a patent (Patents Form 7/77)	NONE
Request for preliminary examination and search (Patents Form 9/77)	NONE
Request for substantive examination (Patents Form 10/77)	NONE
Any other documents (please specify)	NONE

11. I/We request the grant of a patent on the basis of this application.

Signature	Date
R G M SAVIDGE - By Power of Attorney	16.07.99

12. Name and daytime telephone number of person to contact in the United Kingdom

MR R G M SAVIDGE
0121 420 5430

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STRUCTURED SURFACTANT SYSTEMS

The present invention relates to the formulation of structured surfactant suspending systems. It is particularly relevant to the formulation of personal care formulations such as shampoos and skin cleansing preparations.

STRUCTURED SURFACTANT

Suspending solids in liquids presents a problem. If the solids differ in density from the liquid they will tend either to sediment or float. Increasing the viscosity of the liquid can retard, but not prevent such separation, and high viscosities are generally undesirable. Colloidal systems, in which the suspended particles are sufficiently small to experience Brownian motion, e.g. less than 1 micron, may be kinetically stable. However the difficulty or undesirability of comminuting some solids to such sizes, and the impossibility of maintaining many of them at this level in the face of crystal growth or agglomeration, limits the use of colloidal suspensions.

Adjusting the density of one phase to match that of the other is usually impracticable. Moreover such systems are almost always temperature-unstable due to differential rates of thermal expansion.

One method of suspension which permits even relatively large particles to be stably suspended is structured surfactant. The term covers systems in which a surfactant mesophase, usually a lamellar or G-phase, alone or more usually interspersed with an aqueous phase, provides a yield stress which is sufficient, when the system is at rest, to immobilise any suspended particles, but which is sufficiently low to allow the system to be poured like a normal liquid. Such systems may display very low apparent viscosities when stirred, pumped or poured and yet be capable of maintaining particles, sometimes of millimetre or larger size, indefinitely in suspension.

Three main types of suspending system have been employed in practice, all involving a G-phase, in which bilayers of surfactant are arranged with the hydrophobic part of the molecule on the interior and the hydrophilic part on the exterior of the bilayer (or vice versa). The bilayers lie side by side, e.g. in a parallel or concentric configuration, sometimes separated by aqueous layers. G-phases (also known as L_α phases) can usually be identified by their characteristic textures under the polarising microscope and/or by x-ray diffraction, which is often able to detect evidence of lamellar symmetry. Such evidence may comprise first, second and sometimes third order peaks with d-spacing ($\frac{2\pi}{Q}$ where Q is the momentum transfer vector) in a simple integral ratio 1:2:3. Other types of symmetry give different ratios, usually non integral.

Most surfactants form a G-phase either at ambient or at some higher temperature when mixed with water in certain specific proportions. However such G-phases cannot usually be used as structured suspending systems. Useful quantities of solid render them unpourable and smaller amounts tend to sediment.

The main types of structured system used in practice are based on dispersed lamellar, spherulitic and attenuated lamellar phases. Dispersed lamellar phases are two phase systems in which the surfactant bilayers are arranged as parallel plates to form domains of G-phases which are interspersed with an aqueous phase to form an opaque gel-like system. They are described in EP O 086 614.

Spherulitic phases comprise well defined spheroidal bodies, usually referred to in the art as spherulites, in which surfactant bilayers are arranged as concentric shells. The spherulites usually have a diameter in the range 0.1 to 15 microns and are dispersed in an aqueous phase in the manner of a classical emulsion, but interacting to form a structured system. Spherulitic systems are described in more detail in EP O 151 884.

Many structured surfactant systems are intermediate between dispersed lamellar and spherulitic, involving both types of structure. Usually systems having a more spherulitic character are preferred because they tend to have lower viscosity. A variant on the spherulitic system comprises prolate or rod shaped bodies sometimes referred to as batonets.

A third type of structured surfactant system comprises an attenuated G-phase. It differs from the other two types of structure system in being essentially a single phase, and from conventional G-phase in having a wider d-spacing. Conventional G-phases have a d-spacing of about 5 to 7 nanometers. Attempts to suspend solids in such phases results in stiff pastes which are either non-pourable, unstable or both. Attenuated G-phases with d-spacing between 8 and 20, e.g. 10 to 15 nanometers, form when the electrolyte is added to aqueous surfactants at concentrations just below those required to form a normal G-phase, particularly to surfactants in the M phase. The M phase comprises surfactant molecules arranged to form cylindrical rods of indefinite length. It exhibits hexagonal symmetry and a distinctive texture under the polarising

microscope. Typical M phases have so high a viscosity that they appear to be curdy solids. M phases near the lower concentration limit (the L_1/M phase boundary) may be pourable but have a very high viscosity and often a mucous-like appearance. Such systems tend to form attenuated G-phases particularly readily on addition of sufficient electrolyte.

Attenuated G-phases are described in more detail in EP O 530 708. In the absence of suspended matter they are translucent, unlike dispersed lamellar or spherulitic phases which are necessarily opaque. They are optically anisotropic and have shear dependent viscosity. In this they differ from L_1 phases which are micellar solutions and which include microemulsions. L_1 phases are clear, optically isotropic and substantially Newtonian. They are unstructured and cannot suspend solids. Some L_1 phases exhibit small angle x-ray diffraction spectra which show evidence of hexagonal symmetry. Such phases usually have concentrations near the L_1/M phase boundary and may form attenuated G-phases on addition of electrolyte.

Most structured surfactant systems require the presence of electrolyte as well as surfactant and water in order to form structured systems capable of suspending solids. However certain relatively hydrophobic surfactants such as isopropylamine alkyl benzene sulphonate can form spherulites in water in the absence of electrolyte. Such surfactants are capable of suspending solids in the absence of electrolyte as described in EP O 414 549.

APPLICATION

Structured surfactants have been applied to the problems of suspending: water insoluble or sparingly soluble builders in laundry detergent; antifoams and enzymes in laundry detergents and other surfactant systems; abrasives in hard surface cleaners; pesticides and oils in agrochemical preparations (EP O 388 239 and EP O 498 231); rock cuttings in drilling muds (EP O 430 602); dyestuffs in dyebath concentrates and printing inks (EP O 472 089); talcs, oils and other cosmetic ingredients in personal care formulations.

FLOCCULATION

A problem with the two phase structured surfactant systems, and especially spherulitic systems, is flocculation of the dispersed surfactant structures. This tends to occur at high surfactant and/or high electrolyte concentration. It can have the effect of making the composition very viscous and/or unstable with the dispersed surfactant separating from the aqueous phase.

Certain amphiphilic polymers have been found to act as deflocculants of structured surfactants. One type of deflocculant polymer exhibits cteniform (comb-shaped) architecture with a hydrophilic backbone and hydrophobic side chains or vice versa. A typical example is a random copolymer of acrylic acid and a fatty alkyl acrylate. Cteniform deflocculants have been described in a large number of patents, for example WO-A-9106622.

In our copending British patent application no. 9914673 we have described the use of small amounts of carbohydrates such as sugars and alginates as deflocculants.

THE PROBLEM

Existing structured surfactant formulations are constrained by several limitations which have hitherto limited their application, especially in the areas of cosmetics and personal care. These include the following:-

1. Unless a substantial amount of electrolyte is present the choice of surfactant is limited to a fairly small range of relatively insoluble surfactants such as isopropyl alkyl benzene sulphonates.
2. Spherulitic or dispersed lamellar structured surfactants are opaque.
3. Attenuated G phases are opalescent and are usually formed over narrow concentrations and/or temperature ranges which make them difficult to use in practice.
4. At high surfactant concentrations, e.g. above 25% by weight it is difficult to make stable structured systems without using expensive deflocculants and auxiliary stabilisers.

There is a need, especially in the personal care field, for a suspending system that is clear, transparent and mobile. There is a need for a system which contains high levels of surfactant but which does not require expensive deflocculants. There is a need for a system that contains relatively soluble surfactants but which does not require the presence of electrolyte as a structurant.

THE SOLUTION

We have now discovered that formulations meeting some or all of the above needs, may be obtained by using water soluble carbohydrate as a structurant.

THE INVENTION

Our invention provides the use of water soluble carbohydrates as structurants in structured surfactant suspending systems.

According to a second embodiment our invention provides a structured surfactant composition having suspending properties which comprises a surfactant, water and a structurant characterised in that said structurant is a water soluble carbohydrate. The structured composition is typically an attenuated G-phase.

PROPORTIONS

Preferably the surfactant is present in an amount of at least 5%, e.g. at least 10%, especially at least 15% by weight of the composition but preferably less than 60% e.g. less than 50%, especially less than 40%.

Preferably the carbohydrate is present in a proportion of at least 25% e.g. at least 40% and usually more than 40% by weight. Concentrations greater than 65% are usually avoided. Typically the carbohydrate is less than 60%, usually less than 55% by weight of the composition.

The proportion of water is usually greater than 20% by weight, more commonly greater than 30%, typically greater than 40% of the composition, but is preferably less than 65% usually less than 60%, e.g. less than 55%.

THE CARBOHYDRATE

The preferred carbohydrates are mono and disaccharide sugars such as sucrose, glucose or fructose. Other sugars which be used include mannose, ribose, galactose, allose, talose, gulose, idose, arabinose, xylose, lyxose, erythrose, threose, acrose, rhamose and cellobiose. The carbohydrate may be a tri- or tetra-saccharide or a water soluble polysaccharide such as soluble starch. The term "carbohydrate" as used here includes water soluble non-surfactant derivatives of carbohydrates such as carboxylic acids and their salts, e.g. gluconic acid, mannamic acid, ascorbic acid and alginates or reduced sugars such as sorbitol or inositol.

THE SURFACTANT

The surfactant preferably comprises C_{8-25} alkyl or alkenyl groups and a hydrophilic group with at least one alcoholic hydroxyl group and/or ether linkage, such as a non-ionic ethoxylate especially an alcohol ethoxylate, fatty acid ethoxylate or fatty ester ethoxylate, an anionic ethoxy derivative, e.g. an ether sulphate such as C_{10-20} alkyl 1 to 20 mole ethoxy sulphate, a mono or diethanolamide.

The surfactant may additionally or alternatively comprise other anionic, non-ionic or amphoteric surfactants, preferably as minor components of the total surfactant.

ELECTROLYTE

The presence of electrolyte is not required for structuring but is generally tolerated if required for other purposes. We particularly prefer electrolyte-free or low electrolyte (e.g. 1 to 5% by weight) compositions for personal care applications but can tolerate much higher levels, e.g. up to 20% or more if required. For example industrial cleaning formulations may require high levels of alkali such as sodium hydroxide, carbonate or silicate. The presence of builders such as citrate, potassium pyrophosphate, or sodium tripolyphosphate may also be tolerated.

SUSPENDED MATTER

The composition may contain suspended solid, liquid or gaseous particles. For instance the composition may contain suspended oil droplets. The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether) or a fatty glyceride, a terpene oil such as limonene or a silicone oil. Mixtures of oils may be used. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil. Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolved in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids may be suspended including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may also be suspended. Other active ingredients which may be suspended include

insect repellents and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

OTHER INGREDIENTS

The composition may contain minor amounts of other ingredients such as dyes, perfumes, soil suspending agents or optical brighteners. Solvents such as ethanol or isopropyl and hydrotypes such as C₁₋₆ alkyl benzene sulphonates or urea are generally undesirable and are preferably absent but may be tolerated in small amounts, preferably less than 10%, e.g. less than 5%, most preferably less than 2%.

The invention will be illustrated by the following examples.

Example 1

A composition comprising 25% sodium C₁₂₋₁₄ mole ethoxy sulphate, 50% sucrose, 25% water is a clear, transparent, mobile attenuated G-phase with strong suspending properties.

Example 2

The following formulations were prepared:-

	1	2	3	4
C ₁₂₋₁₄ 3 mole ether sulphate	10	12.5	15	17.5
Sucrose	46	46	46	46
Trisodium citrate	2	2	2	2
Perfume	5	5	5	5
Water	balance	balance	balance	balance

All four formulations were clear or slightly hazy, mobile, structured liquids with good suspending properties.

Suspension of talc/and or mineral oil were stable.

Banker Breiten

2/8/2000

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